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### ARNOLD & PORTER

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### September 3, 2003

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Herewith

For:

Improved Methods for Producing Ethanol from Biomass

Applicants:

Ludz WILKENING et al.

Atty Docket:

19003.002

Sir:

The following documents are forwarded herewith for appropriate action by the U.S. Patent and Trademark Office (PTO):

- 1. Provisional Application Transmittal (PTO/SB/16);
- 2. U.S. Provisional Application entitled:

Improved Methods for Producing Ethanol from Biomass and naming as inventors:

Ludz WILKENING and Carl Ludwig WILKENING; the application consisting of:

- a. a specification containing:
  - (i). 9 pages of a description prior to the claims;
  - (ii). 3 pages of claims (25 claims);
  - (iii). 1 pages of drawings (Figure 1);
  - (iv). a one (1) page abstract; and
- 3. two (2) return postcards.

### ARNOLD & PORTER

Commissioner for Patents Atty. Docket: 19003.002 Ludz WILKENING et al. Page 2

Please stamp one of two attached postcards with the filing date of these documents and return it to our courier, and stamp the other prepaid postcard with the filing date and unofficial application number and return it as soon as possible.

Applicants request that the following fees be charged to Deposit Account No. 50-2387 referencing docket number 19003.002:

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Applicants do not believe that any fees other than the above fees (\$80.00) are due in conjunction with this filing. However, should any additional fees be required, the Patent and Trademark Office is hereby authorized to charge any fee deficiency, or credit any overpayment, to our Deposit Account No. 50-2387 referencing docket number 19003.002. A duplicate copy of this letter is enclosed.

Very truly yours,

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**Attachments** 

### Improved Methods for Producing Ethanol from Biomass

The present invention relates to improved methods for producing ethanol from biomass, comprising

- (a) reducing the size of the biomass to a particle size of less than 1 mm;
- (b) utilizing the remaining biomass as a medium for fermentation; and
- (c) obtaining the ethanol;

wherein the proteins of the biomass and the bran optionally present are substantially separated from the biomass prior to step (b) or (c).

The pulp obtained by fermentation can be used for obtaining a high-quality feeding stuff. The clear phase of the pulp can be utilized for obtaining methane gas for energy production and for conversion in a combined heat and power production of energy and steam.

The production of alcohols via fermentation of biomass represents one of the oldest biotechnological methods. It is used inter alia for the production of alcoholic beverages, such as beer and wine. Similarly, the production of alcohol for industrial use by fermentation of biomass is also known for a long time. The respective alcohols, in particular ethanol, are presently used as starting material for the preparation of pharmaceutical compositions, cosmetics and a number of chemicals.

Likewise, the use of ethanol obtained by fermentation as an energy carrier is known for a long time. However, due to the high costs compared to the exploitation of crude oil, it was not commercially employed in the past.

On account of the reduced availability of crude oil reserves and the CO<sub>2</sub>-reduction requested in the Kyoto-Treaty, the possible use of bioethanol as an energy carrier attracted new interests. Consequently, there is a need for improved methods for the production of ethanol from biomass in large scale.

Several processes for the production of alcohol from biomass are described in the state of the art. Since entirely different biomasses are used in these processes as starting materials, inter alia sugarcane, sugar beet, grain etc., the processes for obtaining bioalcohols also differ from each other. Although identical starting materials may be used, different protocols for carrying out the process exist.

The fermentation processes for producing ethanol from grain all comprise

- (a) reducing the size of the grain;
- (b) adding microorganisms to the biomass and carrying out a fermentation; and
- (c) separating the ethanol from the biomass.

The separation of the ethanol is usually achieved by distillation. In this step pulp is obtained as a side product and is usually concentrated and used as fertilizer or (after drying) as a protein-containing feeding stuff.

Each of these steps is described in many different embodiments in the state of the art. The process presently used in the art at is described for example in DE 30 07 138, hereby incorporated by reference. This document discloses a process for the production of ethanol by fermentation of a carbohydrate-containing substrate in one or more fermentation vessels. After fermentation, the fermentation fluid is distilled, rectified, and – if necessary – dehydrated to water-free alcohol. The state of art further teaches to divide the flow of fermentation fluid in at least one flow of yeast concentrate and one yeast-free flow. The yeast concentrate flow is re-circulated into the fermentation vessel and the yeast-free phase is divided into an ethanol-rich flow and a rest flow (pulp) by means of distillation. The rest flow is partially re-circulated into the fermentation vessel. The advantages of the process described in DE 30 07 138 reside in the fact that the yeast-free flow can be processed in a simple evaporator using one or a few distillation steps, by dividing the yeast-free flow into an ethanol-rich first steam flow and a first fluid bottom flow. The steam flow is transferred into a production plant for producing the desired ethanol quality. The bottom flow is partially re-circulated into the fermenter, while another part is lead to a depression unit, wherein this part is divided into an ethanol-rich second steam flow and a second fluid bottom flow, which has a low concentration of ethanol.

The ethanol-rich steam flow can be used together with the first ethanol-rich steam flow for obtaining ethanol, for example in a rectification column.

Since the pulp contains large amounts of proteins, it is usually used as feeding stuff. However, the production of feeding stuff and in particular the drying of the pulp requires a lot of energy, approximately 50 % of the energy needed for the entire bioethanol production process.

Furthermore, methane gas can be obtained from the pulp and the rest can be used as a fertilizer. For example, ZA80/5297, hereby incorporated by reference, discloses a process in which ethanol is obtained from maize and the pulp is used for producing methane. For this purpose, the maize is reduced in size, bran and proteins are separated, and a substrate for fermentation is obtained. The pulp generated by fermentation and distillation is to be used for obtaining methane gas. However, the device used for reducing the size of the corn, a swing-hammermill, usually generates fragments of comparatively large size with a particle size of approximately 2 mm. However, the size of these particles makes methane production more difficult.

All processes known in the art share the problem that a long retention time is required for degassing the pulp. As a result, the whole production plant will become expensive since a very large volume is required. It was therefore not possible to construct bioethanol production plants producing more than 300 m<sup>3</sup> ethanol per day which obtain methane from the pulp for energy production.

To solve this problem, the present invention provides processes for the production of ethanol from biomass comprising:

- (a) reducing the size of the biomass to a particle size of less than 1 mm;
- (b) utilizing the remaining biomass as a medium for fermentation; and
- (c) obtaining the ethanol;

wherein proteins present in the biomass and the bran optionally present are substantially separated prior to step (b) or (c).

According to the invention, it was surprisingly shown that carrying out the fermentation and/or the distillation using a substrate which was reduced to a particularly small particle size and does not contain bran or proteins of the biomass has considerable advantages in the subsequent distillation and processing. The lower content of solids and the smaller size of the remaining content of solids in the alcoholic mash results in advantages during distillation and considerable savings in energy and time in processing the pulp after distillation. The pulp with a reduced content of solid matter is particularly suitable for obtaining methane. In particular, the retention time of the cleared pulp in the methane production plant is considerably reduced when compared to the retention time of common pulp, which allows a considerable reduction of the size of the plant.

According to a preferred embodiment of the invention, the process of ethanol production therefore directly generates energy and/or heat which can be utilized for the process (distillation, rectification and/or drying of the solid phase of the pulp).

The feeding stuff obtainable from the protein, the bran and the solids of the pulp by the process of the invention is of a higher quality compared to feeding stuff produced by methods common in the art, since the feeding stuff of the invention has only been exposed to a mild heat treatment.

According to the invention, the process has particular advantages in the production of ethanol starting from grain as biomass. In particular, wheat, rye or triticales are used. The use of wheat is especially preferred.

In the following, the individual process steps illustrated in detail:

First, the size of the biomass is reduced. For this purpose, any process for reducing the size of biomasses can be used. Numerous such processes are known in the state of the art. In particular, the size of the biomass can be reduced by milling. Reducing the size of the biomass to a granule size of less than 1 mm is preferred, wherein reducing to a particle size of less than 0,5 mm or to approximately 0,2 mm is particularly preferred. According to the invention, the particle size can be selected by sieves.

Prior or subsequent to the size reduction, the biomass can be subjected to swelling by the addition of liquid, in particular by addition of water. Preferably, the biomass is milled in a dry condition, since the bran can be separated more easily in this state. Any method known in the state of the art can be used for the separation of the bran. Usually, the bran is separated by sieves.

Subsequently, the biomass is liquefied by the addition of enzymes and saccharified, whereby a substrate is obtained. If further liquefaction is desired, water and other auxiliary agents can be additionally added.

In the processes according to the invention, proteins are further removed from the substrate prior to the Initiation of fermentation. Proteins can be removed according to methods known in the state of the art, a precipitation of proteins by chilling the substrate is particularly preferred. The precipitated proteins can subsequently be separated and dried.

In this manner, a substrate with a high content of starch and a low content of solids of 3 to 50 %, and preferably with a content of solid matter of 6 to 10 %, can be obtained. The determination of the solid content is performed by centrifugation. Due to the substrate's low content of solids, it is possible to perform a particularly advantageous separation process after distillation.

During fermentation, the carbohydrates present in the biomass are fermented by the addition of microorganisms, thereby producing alcohol, in particular large amounts of ethanol. Usually, yeasts are

added for the initiation of fermentation. The fermentation is usually carried out according to methods known in the state of the art. Preferably, yeasts are used for fermentation and the fermentation stops, when substantially all carbohydrates are have been transformed by fermentation.

An alcoholic mash is obtained by fermentation. The alcohol present in the mash can be distilled and further purified by methods known in the state of the art. The known methods for distillation, rectification and dehydration can be employed. By the separation of bran and proteins according to the invention, less side-products of ethanol are generated during fermentation. This leads to a high-quality crude alcohol, which reduces the expense for rectification. The efficiency of the process according to the invention is thus improved by the use of the alcoholic mash having a lower content of solids.

According to the process of the invention, the pulp obtained by distillation can subsequently be chilled and divided into a solid phase and a clear phase. After distillation, the pulp has a temperature of approximately 100°C and a milky color. After chilling, the pulp has a temperature of approximately 30 to 40°C and contains solid matter which is precipitated during chilling. This solid matter can be separated according to the invention, for example by use of a separator, preferably a disk separator.

The solid phase of the pulp can be used for the production of feeding stuffs or fertilizers. The clear phase has a content of solids of less than 10 %, preferably clearly less than 10 %, such as less than 5 % or 1 %, wherein obtaining a clear phase with a content of solids atter of less than 0,5 % is particularly preferred.

This clear phase can be used for obtaining methane according to processes known in the state of the art. The use of a clear phase with a lower content of solid matter in this step of the procedure has the particular advantage that methanation of the clear phase of the pulp can be performed in a so-called high-performance reactor. An respective reactor works with beads which usually have a diameter of 1 to 2 mm and in which methane bacteria are immobilized. Thereby, a multiple, preferably at least a 5-fold space time yield can be obtained. Usually, the beads are sponge-like and consist of lime. By use of these beads, the efficiency of the reactor is considerably increased compared to common reactors. The use of these reactors for the first time renders possible the construction of a particularly advantageous bioethanol plant, which comprises methanation of the pulp and has a very high daily capacity, in particular a daily capacity of more than 300, preferably more than 500 m<sup>3</sup> ethanol per day.

The reactor can be preceded by a mixing and compensation vessel which allows a qualitatively and quantitatively constant supply of clear phase to the methanation plant. By circulation pumps, an intensive fluid exchange between the two vessels is achieved.

In the lower part and the middle part of the reactor, the methane bacteria beads can be kept suspended by the ascending gas and additional pumping. In this manner an intensive mixing of the reaction material is achieved, which further increases the efficiency. Preferably, collectors are located in the upper part of the reactor, which retain the immobilized methane bacteria lime beads in the reactor.

The content of solids present in the substrate is entrained by the liquid flow; after deposition they leave the system together with the purified substrate. If a higher content of solids is used, the solids will accumulate in the methane reactor, thus leading to a stop of agitation of the immobilized methane bacteria and a stop of the entire process. Extensive studies with a technical plant have shown that the process can be carried out with particular advantages in this step, when the content of solids of the clear phase of the pulp is 0,5 % or less.

The methane can subsequently be converted to energy or heat according to methods known in the art, whereby energy and process heat are generated which cover the energy need of the conversion.

According to a particular preferred embodiment, the invention therefore relates to a process for the production of ethanol from grain, comprising

- (a) milling the grain to a particle size of less than 1 mm and separating the bran from the flour using a sieve system;
- (b) enzymatically liquefying and purifying the flour in a common manner including the addition of water, thereby obtaining a mash;
- (c) substantially precipitating the proteins present in the mash by chilling, sieving and drying, thereby obtaining the proteins and a substrate;
- (d) fermenting and distilling the substrate in a common manner, thereby obtaining ethanol and a pulp;
- (e) dividing the pulp in a solid phase and a clear phase, thereby obtaining a clear phase with a content of solids of less than 1 %; and
- (f) obtaining methane from the clear phase in a high-performance methane reactor.

The solid phase of the pulp can be mixed with the solid phases previously obtained, namely with the proteins and the bran. It is preferred that the mixture obtained in this manner is subsequently dried under mild temperatures. Thereby a feeding stuff is obtained which has a higher quality compared to feeding stuffs obtained by methods for the production of alcohol from biomass known in the art.

The process according to the invention is shown in Figure 1. Figure 1 illustrates the process according to the invention as presently conducted in an experimental production plant. However, it should be

understood from the above description that not all steps which are illustrated in Figure 1 are necessary for obtaining the advantages of the invention.

As shown in Figure 1, grain from a grain stock is provided. Subsequently, the grain is milled and the bran is separated.

By the addition of enzymes, auxiliary substances and water, the grain is liquefied and subsequently saccharified. The proteins present in the saccharified substrate are precipitated by chilling and separated. A substrate is obtained which comprises all of the starch in the grain. In this manner, the components which do not produce alcohol are separated prior to the fermentation process.

This substrate serves as a medium for fermentation. For this purpose, yeast, nutrients, such as ammonium sulfate and bitter salt, air and water are added.

The fermentation produces an alcoholic mash which is employed in a distillation for the production of a crude alcohol according to methods known in the art. In the distillation, crude alcohol is produced which is purified by rectification and dehydration to bioethanol or a neutral alcohol. The purity of the product obtained can be between 96 and 99,9 vol-%. If fusel oils, singlings, pre- and last runnings are produced during this procedure, these can be used for the production of methane.

As a side-product of the distillation, pulp is obtained, which is first chilled and subsequently divided into a solid phase and a liquid phase. The liquid phase is introduced into the methanation production plant. The methane obtained is converted to energy, and process heat is obtained.

The solid phase obtained from the separation of the pulp is mixed with the bran and the proteins and subsequently dried. Thereby, a protein-containing feeding stuff is obtained which is suitable for feeding animals, in particular cows, pigs, horses, etc.

A trial plant, which corresponds to the system depicted in Figure 1, has been run for months and has been used batch-wise for the production of ethanol, wherein 100 I ethanol were obtained from 170 kg starch. Based on these studies, the following energy balance was obtained for the process according to the invention:

#### 1. Quantity balance

Mash containing approximately 10 vol-% ethanol. Therefore, the following quantity balance applies for the mash:

10 m<sup>3</sup>/m<sup>3</sup> ethanol mash: 10 vol-% ethanol

1 m<sup>3</sup>/m<sup>3</sup> ethanol ethanol (pure)

9 m<sup>3</sup>/m<sup>3</sup> ethanol pulp

- Energy content of the clear phase of the pulp suitable for methanation: 2.
- 2.1 Content of the clear phase:

In this section, the chemical oxygen demand (COD) per m<sup>3</sup> ethanol of the clear phase of the pulp according to the invention is described and the portion which is available for energy production by methanation:

burden per m³ waste water 40 kg COD/m<sup>3</sup>

biotechnical availability

80 %

net burden per m³ waste water

32 kg COD/m<sup>3</sup>

The clear phase thus provides carbohydrates for methanation which have a chemical oxygen demand of 32 kg/m³ waste water. The clear phase has an energy content of 100 kWh/m³ waste water.

2.2 From the above, the following energy balance can be inferred in relation to ethanol:

Pulp (normative) = 9 m<sup>3</sup> pulp/m<sup>3</sup> ethanol x energy content = 900 kWh/m<sup>3</sup> ethanol. Corresponds to 3600 MJ/m<sup>3</sup> ethanol.

- In comparison, the process of the invention has an energy requirement which is as follows: 3.
- 3.1 Energy consumption distillation/rectification:
  - steam requirement:  $1800 \text{ kg/m}^3$  ethanol x 2,02 MJ/kg =  $3636 \text{ MJ/m}^3$  ethanol (estimated);
  - energy requirement: 302 kWh/m<sup>3</sup> x 3,6 MJ/kg = 1087 MJ/m<sup>3</sup> ethanol; and
  - total energy consumption = 4723 MJ/m<sup>3</sup> ethanol.
- Energy consumption of drying the pulp: 3.2
  - steam requirement: 1250 kg/m<sup>3</sup> ethanol x 2,02 MJ/kg = 2525 MJ/m<sup>3</sup> ethanol;

energy requirement: 50 kWh/m³ ethanol x 3,6 MJ/kg = 180 MJ/m³ ethanol;

Drying the pulp therefore consumes energy corresponding to a total of 2705 MJ/m<sup>3</sup> ethanol.

- 3.3 Therefore, the total energy consumption (3.1 and 3.2) is:
  - steam requirement of 3050 kg/m<sup>3</sup> ethanol x 2,02 MJ/kg = 6161 MJ/m<sup>3</sup> ethanol; and
  - energy requirement of 352 kWh/m³ ethanol x 3,6 MJ/kg = 1267 MJ/m³;

The total energy consumption therefore is 7428 MJ/m<sup>3</sup> ethanol.

- 4. In contrast, the energy balance of common methods is:
  - steam requirement for the distillation/rectification, dehydration: 2200 kg/m³ ethanol x 2,02 MJ/kg = 4400 MJ/m³ ethanol;
  - energy requirement for the distillation/rectification, dehydration: 302 kWh/m $^3$  ethanol x 3,6 MJ/kg = 1087 MJ/m $^3$  ethanol;
  - steam requirement for drying the grain pulp: 3100 kg/m<sup>3</sup>
     ethanol x 2,02 MJ/kg = 6262 MJ/m<sup>3</sup> ethanol;
  - energy requirement for drying the grain pulp: 178 kWh/m³
     ethanol x 3,6 MJ/kWh = 641 MJ/m³ ethanol;

This corresponds to a total energy consumption of 12434 MJ/m<sup>3</sup> ethanol.

5. It was thus shown that the process according to the invention consumes 5006 MJ/m³ ethanol less energy than the methods known in the art. This corresponds to a reduction of the energy consumption of 40 %.

Further, if the energy production by methanation is added (3600 MJ/m³ ethanol), an improvement of the energy balance of a total of 69 % is obtained. In other words, the process according to the invention requires only approximately 30 % of the energy which is required for carrying out common methods for the production of ethanol from biomass. This impressively demonstrates the advantages of the process according to the invention.

#### Patent claims

- 1. Method for producing ethanol from biomass, comprising
  - (a) reducing the size of the biomass to a particle size of less than 1 mm;
  - (b) utilizing the remaining biomass as a medium for fermentation; and
  - (c) obtaining the ethanol;

wherein proteins present in the biomass and the bran which might be present are substantially separated prior to step (b) or (c).

- 2. Method according to claim 1, wherein in step (a) the biomass is reduced to a particle size of less than 0,5 mm, preferably to a particle size of 0,2 mm.
- 3. Method according to claim 1 or 2, wherein grains or potatoes are used as biomass, in particular wheat, rye or triticales.
- 4. Method according to any of the preceding claims, wherein the bran is separated after size reduction.
- 5. Method according to any of the preceding claims, wherein the biomass is saccharified by the addition of enzymes after separation of the bran.
- 6. Method according to any of the preceding claims, wherein the proteins present in the saccharified substrate are precipitated by chilling.
- 7. Method according to any of the preceding claims, wherein the precipitated proteins are separated.
- 8. Method according to any of the preceding claims, wherein the separation of the bran and the proteins results in a substrate with a content of solids of 3 to 15 %, preferably 6 to 10 %.
- 9. Method according to any of the preceding claims, wherein fermentation is initiated by the addition of the yeast after separation of the proteins.
- 10. Method according to any of the preceding claims, wherein the alcoholic mash obtained by fermentation is distilled in order to obtain crude ethanol.

- 11. Method according to any of the preceding claims, wherein the crude ethanol is rectified and (if necessary) dehydrated, in order to obtain bioethanol, in particular neutral ethanol.
- 12. Method according to any of the preceding claims, wherein the pulp is divided in a solid phase and a clear phase for methanation.
- 13. Method for producing ethanol according to any of the preceding claims, comprising
  - (a) milling the grain to a particle size of less than 1 mm and separating the bran from the flour using a sieve system;
  - (b) enzymatically liquefying and saccharifying the flour in a common manner under the addition of water, thereby obtaining a mash;
  - (c) substantially precipitating the proteins present in the mash by chilling, sieving and drying, thereby obtaining the proteins and a substrate;
  - (d) fermenting and distilling the substrate in a common manner, thereby obtaining ethanol and pulp;
  - (e) dividing the pulp into a solid phase and a clear phase, wherein a clear phase with a content of solids of less than 1 % is obtained; and
  - (f) obtaining methane from the clear phase in a high-performance methane reactor.
- 14. The process for producing ethanol according to any of the preceding claims, wherein a highperformance methane reactor is employed comprising beads with a diameter of 1 to 2 mm, in which methane bacteria are immobilized.
- 15. Method for producing ethanol according to any of the preceding claims, wherein the immobilization of the methane bacteria in the beads in a reactor for methanation increases the space time yield and preferably allows an at least 5-fold space time yield.
- 16. Process for producing ethanol according to any of the preceding claims, wherein more than 300 m<sup>3</sup>, preferably more than 500 m<sup>3</sup> ethanol per day are produced.
- 17. Method for producing methane, comprising a method for producing ethanol and an alcoholic pulp according to one of the claims 1 to 16.
- 18. Method for producing energy or heat, comprising a method for producing ethanol or an alcoholic pulp according to one of the claims 1 to 17, obtaining methane from the pulp and converting the methane and/or the ethanol to energy and/or heat.

- 19. Use of an alcoholic mash with a content of solids of less than 10 weight-%, preferably less than 5 weight-% or 1 weight-% and more preferably less than 0,5 weight-% for the production of methane, energy and/or heat.
- 20. Use according to claim 19, wherein a high-performance methane reactor is used for the production of methane comprising beads with a diameter of 1 to 2 mm, in which methane bacteria are immobilized.
- Use according to claim 19 or 20, wherein the immobilization of the methane bacteria to the beads in the reactor for methanation increases the space time yield and preferably facilitates an at least 5-fold space time yield.
  - 22. Method for the preparation of a feeding stuff, wherein bran, protein and the solid phase obtained from the pulp according to one of the claims 1 to 16 are isolated, mixed and dried.
  - 23. Ethanol, obtainable by a method according to one of the claims 1 to 16.
  - 24. A feeding stuff, obtainable by a method according to claim 22.
  - 25. Methane obtainable by a process according to claim 17.

#### **Abstract**

The present invention relates to methods for producing ethanol from biomass, comprising

- (a) reducing the size of the biomass to a particle size of less than 1 mm;
- (b) utilizing the remaining biomass as a medium for fermentation; and
- (c) obtaining the ethanol;

wherein the proteins present in the biomass and the bran which, might be present are substantially separated prior to step (b) or (c).

The present invention further relates to methods for producing methane and/or feeding stuffs, comprising the above steps.

